भारतीय मानक
Indian Standard

IS 326 (Part 1) : 2022

प्राकृतिक और संश्लेषित सुगंध सामग्री के नमूने लेने और परीक्षण की पद्धतियाँ

भाग 1 नमूना लेना

(चौथा पुनरीक्षण)

Methods of Sampling and Test for Natural and Synthetic Perfumery Materials

Part 1 Sampling

(Fourth Revision)

ICS 71.100.60

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July 2022 **Price Group 5**

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Fragrance and Flavour Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1952 and subsequently revised in 1968, 1984 and 2018. Till second revision, the standard (indigenous) covered the sampling procedures for both natural and synthetic perfumery materials. However, the third revision being identical adoption of ISO 212 : 2007 'Essential oils — Sampling' issued by the International Organization for Standardization covered sampling of essential oils only. Therefore, the Committee decided to revise it to include sampling of natural as well as synthetic perfumery materials.

The composition of the Committee responsible for the formulation of this Standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard

METHODS OF SAMPLING AND TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 1 SAMPLING

(Fourth Revision)

1 SCOPE

This Indian Standard (Part 1) prescribes methods of sampling for natural and synthetic perfumery materials, for the purpose of determining their organoleptic, physical and chemical characteristics.

2 REFERENCES

The following Indian Standard is necessary adjuncts to this standard. The standard contains provisions which through reference in this text constitute provisions of this standard. At the time of publication, the edition indicated was valid. All Standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent revision of the standard indicated below:

IS No.	*Title*
528 : 1999	Oil of *Mentha Arvensis* — Specification (*third revision*)
5757 : 1992	Oil of pine — Specification (*second revision*)
6597 : 2001	Glossary of terms relating to fragrance and flavour industry (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 6597, and the following shall apply.

3.1 Sampling — The collection of a small portion (called the sample) representative of the properties and composition of the lot of the material sampled.

4 METHOD OF SAMPLING

4.1 General

The difficulties encountered in sampling are often considerable and depend upon such factors as the number and capacity of the containers, the physical state of substance, and the presence of solid, natural constituents and separated impurities. In order to obtain a representative sample, the procedure may have to be varied considerably.

NOTE — Sampling should consequently be entrusted to experienced personnel able to cope with unforeseen circumstances.

4.2 General Requirements of Sampling

4.2.1 *General Precautions*

In drawing, preparing, storing and handling samples the following general precautions and directions shall be observed:

a) Samples shall not be taken in an exposed place;

b) The sampling equipment and the containers for samples shall be made of aluminium or glass on which the material has no action. They shall be clean, dry and free from all odors;

c) Precautions shall be taken to protect the samples, the material being sampled, the sampling equipment and the containers for samples from adventitious contamination;

d) To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both, by suitable means or by rolling;

e) The sample containers shall be of such size that they are almost the sample. The head space shall be but not completely filled by between 5 to 10 percent of the volume of the container depending on the method of transport adopted. For pasty and semi-solid substance, the containers shall have a wide neck;

f) Each sample container shall be sealed air-tight after filling and marked with full identification particulars, such as the sample number, the date of sampling, the month and year of manufacture of the material and any other relevant particulars of the consignment;

g) Samples shall be stored in a cool place and protected from light and excessive variations of temperature. If agreed to, samples may also be stored-in an inert atmosphere; and

h) Avoid spillage and personal contact and have appropriate clean-up kits during sampling. Use appropriate and recommended personal protective equipment while carrying out sampling.

4.2.2 *Additional Precautions*

The following additional precautions shall be observed:

a) Rubber stoppers or composition corks shall not be used for closing the sample bottles;

b) Samples shall be protected by covers or oil-proof plastics, aluminium or tin foil or any other suitable impervious material over the stopper to keep the moisture and dust away from the mouth of the bottle and to protect it while being handled;

c) To avoid spoilage of the samples under no circumstances, shall sealing wax be applied direct to the cork;

d) All samples shall be transported with special care; and

e) Samples for analysis shall be sent to the concerned laboratory as soon as possible after they have been taken.

4.3 Sampling Equipment

4.3.1 The recommended forms of sampling equipment are:

a) closed type sampling tubes, undivided or divided, for sampling non-homogenous liquids, semi-liquids, free-flowing powders, flasks and pastes;

b) open type sampling tube, for sampling homogenous liquids; and

c) Sampling scoop for sampling materials in the form of powder, crystals, tablets and lumps broken into pieces, if necessary.

4.3.2 *Closed Type Sampling Tube, Undivided* (*see* Fig. 1)

Consists of two concentric metallic tubes closely fitted into each other throughout their entire length, so that one tube can be rotated within the other. Longitudinal opening of about one-third the circumference are cut in both tubes.

In one position, the openings in the two tubes coincide; the sample tube is open when in this position and admits the material. By turning the inner tube through an angle of 180°, it becomes a sealed container. The inner tube may have a diameter of 20 to 40 mm and is undivided along its length to serve as a single container.

The two concentric tubes shall be provided with V-shaped ports at their lower ends, so placed that the material contained in the equipment can be drained through them, when the longitudinal openings are in line.

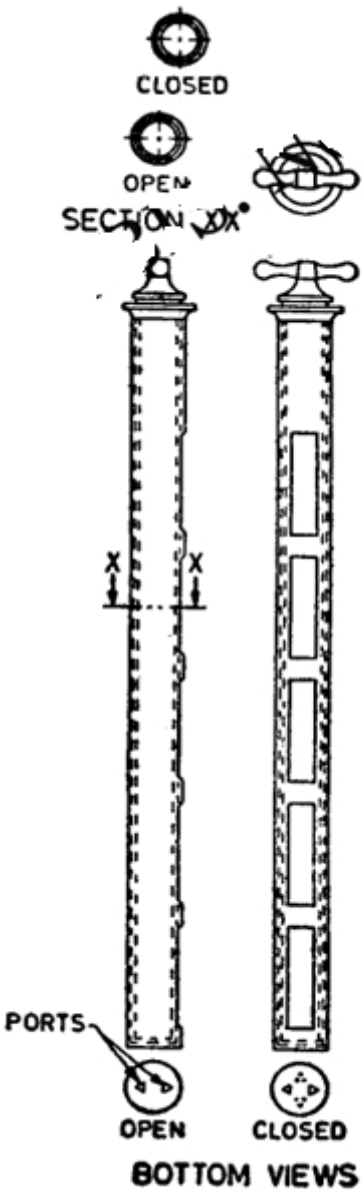

FIG. 1 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

The length of the equipment shall be, such as to enable it to reach the bottom of the container being sampled.

The equipment is inserted closed, the material is admitted by opening it, and finally it is closed and withdrawn.

4.3.3 *Closed Type Sampling Tube, Divided* (*see* Fig. 2)

It is also of metal and has D-shaped cross section. It is provided with compartments along its length and is opened and closed by means of a closely fitting shutter which moves up and down throughout the entire length. It may be from 25 to 50 mm wide.

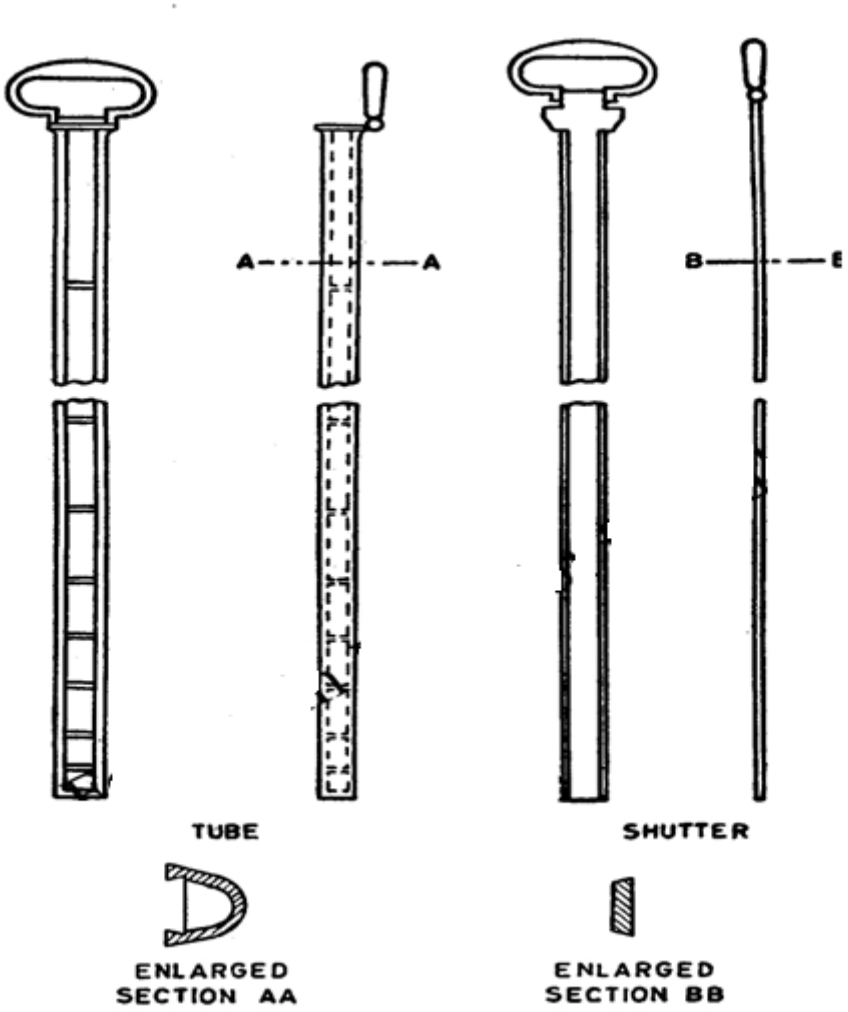

FIG. 2 CLOSED TYPE SAMPLING TUBE, DIVIDED

The equipment is inserted closed, the shutter is pulled out to admit the material, and the tube then closed and withdrawn.

4.3.4 *Open Type Sampling Tube for Homogeneous Liquids* (*see* Fig. 3)

It is made of metal or thick glass, and may be of 20 to 40 mm diameter and 400 to 800 mm length. The upper and lower ends are conical and narrow down to 5 to 10 mm diameter. Handling is facilitated by two rings at the upper end. For taking a sample, the equipment is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

4.3.5 The sampling scoop made of a suitable metal shall be as shown in Fig. 4.

4.4 Sampling

4.4.1 Sampling of large capacity containers (tanks, tank cars, etc).

4.4.1.1 Five partial samples shall be withdrawn from each container, at depths from the upper surface approximately equal to:

a) 10 percent of the total depth;
b) one-third of the total depth;
c) half of the total depth;
d) two-thirds of the total depth; and
e) 90 percent of the total depth.

For each container, the five partial samples shall be bulked and homogenized. From this bulk three representative samples shall be drawn.

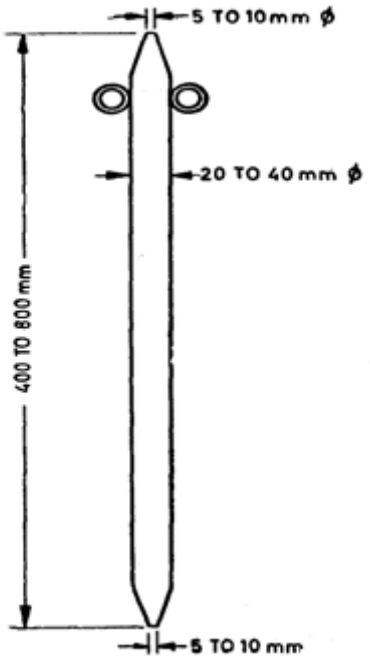

FIG. 3 OPEN TYPE SAMPLING TUBE

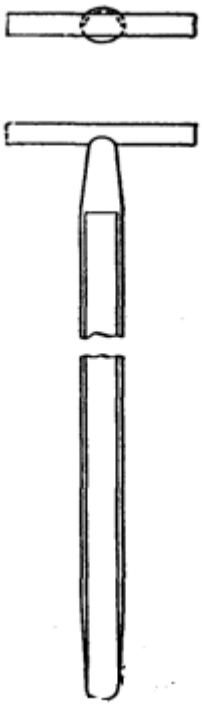

FIG. 4 SAMPLING SCOOP

The quantity of the material for each of the three samples shalt be sufficient to make triplicate determination of all the requirements given in the specification.

4.4.1.2 Should there be impurities or water at the bottom or at the surface of the essential oil, a sample of such a layer shall be withdrawn separately, thoroughly mixed and divided into three similar portions (*see* **4.5.1**). These specimens shall not be added to the five partial samples referred to above, but shall be retained and marked separately so that the nature of the impurities can be ascertained.

4.4.1.3 For each container, a small but approximately equal quantity of material shall be taken from the three samples selected according to **4.4.1.1** and thoroughly mixed so as to constitute the composite sample for the container. The quantity of material for the composite sample shall be sufficient to make the triplicate determination of oil the characteristics tested on the composite sample.

4.4.1.4 All the-individual and composite samples shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third to be used as a referee sample. For packing of individual and composite sample the procedure given in **4.5.3.2** and for referee test samples the procedure given in **4.5.3.3** shall be followed.

4.4.2 Sampling of other containers (drums, jugs, carboys, flasks, bottles, etc).

4.4.2.1 *Lot*

In a single consignment of one type and grade of material, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of one type and grade of material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

4.4.2.2 For ascertaining the conformity of the lot to the requirements of material specifications, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (*n*) shall depend on the size of lot (*N*) and shall be in accordance with Table 1.

If inspection reveals no impurities or water, one partial sample shall be withdrawn from each container, the contents having first been homogenized.

If inspection reveals the presence of impurities or water near the bottom or at the surface, specimens containing such foreign matter shall be withdrawn separately from each container, as specified under **4.4.1.2**, such specimens shall be kept and marked separately. More

than one partial sample may have to be withdrawn from the bulk, the number of such samples and the depth from which they are withdrawn depending upon the size of the container. Partial samples shall not be withdrawn from distances from the upper surface or bottom less than 10 percent of the total depth.

In every case, the partial samples taken from all the containers shall be bulked and homogenized.

From this bulk three representative samples shall be drawn.

Table 1 Scale of Sampling

(*Clauses* 4.4.2.2 *and* 4.4.2.3)

Size of the Lot (*N*) (Number of Containers)	Minimum Number of Containers to be Selected (*n*)
1 to 3	Each container
4 to 20	3
21 to 60	4
61 to 80	5
81 to 120	6
121 and above	One in every twenty

4.4.2.3 The containers shall be selected at random. In order to ensure the randomness of selection, reference may be made to IS 4905. In case this standard is not readily available following procedure may be adopted:

a) If the lot consists of individually packed containers, then starting from any container in the tot, count them in any suitable order as 1, 2, 3 . . . . . up to *r* and so on where *r* is the integral part of *N*/*n*. Every rth container thus counted shall be withdrawn till the requisite number of containers is obtained.

b) If the lot consists of cartons or cardboard boxes each containing more than one container, then a certain number of boxes (not less than 5 percent of the total number of boxes in the lot wherever feasible) shall be chosen first. From each of the boxes so chosen, approximately equal number of containers shall then be selected so as to obtain the required number of containers specified in Table 1.

4.4.2.4 From each of the containers selected according to **4.4.2.3** a small representative portion of the material shall be drawn with the help of the sampling equipment. The approximate quantity of material to be drawn from a container shall be nearly equal to thrice the quantity required for testing indicated in **4.6**.

4.5 Preparation of Test Samples (Procedure)

4.5.1 *Inspection*

The first operation of the sampling procedure is inspection of the consignment.

The physical consistency of essential oils can be one of the following:

a) Liquid (occurring most frequently),

b) Solid,

c) A mixture of, liquid and solid, and

d) Pasty.

It is desirable, where possible, to ascertain whether the material in each of the containers of the lot is uniform in appearance and, in the case of liquid, whether any or all the lots contained separated solids, water or other impurities. When, owing to the nature of the container, this cannot be done directly, portions of the material shall be withdrawn by means of a proper equipment (*see* **4.3**), so that specimens from the surface and from the bottom can be examined. If the container has a cock or bung hole at the bottom, specimens from the lower part may be withdrawn through this.

4.5.2 *Homogenization*

It is necessary to ensure that the sample taken from each container is a fair average of its contents. Homogenization is obtained as follows.

4.5.2.1 In the case of liquid products, it is sufficient to shake the container, to use some form of agitator, or to homogenize by injection of nitrogen or de-oxygenated air.

4.5.2.2 In case of materials in a solid or pasty condition or composed of mixed solid and liquid phases, these are mixed, when possible, by shaking and by exposing the container to a higher temperature or by warming it artificially until the contents are liquefied. The maximum limit of the heating temperature, if necessary 'will be specified in the individual product specification.

When total liquefaction cannot be attained, a series of partial samples shall be withdrawn by means of an appropriate equipment (*see* **4.3**) and in the manner indicated in **4.4**. The partial samples shall be bulked and homogenized.

4.5.2.3 In the case of materials in the form of powders, crystals, tablets or lumps which cannot be liquefied, the material shall be drawn from different parts of the containers with the help of a suitable sampling equipment so as to give a representative sample for the container.

NOTE — In case the material to be taken out from a container is not adequate for testing purposes, the requisite quantity of the material may be made up from additional containers.

4.5.3 Out of these portions, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics specified under **4.6.1**. The composite sample shall be divided

into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

4.5.3.1 The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample, one set of individual samples representing the *n* containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as referee sample.

4.5.3.2 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars.

4.5.3.3 The referee test samples consisting of a composite sample and a set of *n* individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed between the purchaser and the supplier to be used in the case of any dispute between the two.

4.6 Number of Tests

Tests for the determination of important characteristics, as specified in the relevant product specifications, shall be conducted on each of the individual sample separately.

4.6.1 Tests for the determination of all the remaining characteristics in the product specification shall be conducted on the composite sample.

4.7 Criteria for Conformity

4.7.1 *For Individual Samples*

For each of those characteristics, which have been determined on the individual samples, the mean ($\bar{x}$) and range (R) of test results shall be calculated as follows:

$$\text{Mean } (\bar{x}) = \frac{\textit{The sum of test result}}{\textit{Number of test results}}$$

Range (R) = The difference between the maximum and the minimum values of the test results.

4.7.1.1 If the specification limit for the characteristic is given as a minimum, then the value of the expression $[(\bar{x}) - KR]$ shall be calculated from the relevant test results (*see* **4.7.1.4**). If the value so obtained is greater than or equal to the minimum limit, the lot shall be declared as conforming to the requirement of that characteristic.

4.7.1.2 If the specification limit for the characteristic is given as a maximum, then the value of the expression $[(\bar{x}) + KR)]$ shall be calculated from the relevant test results (*see* **4.7.1.4**). If the value so obtained is less than or equal to the maximum limit, the lot shall be declared as conforming to the requirement of that characteristic.

4.7.1.3 If the characteristic has bilateral specification limits, then the values of the expression $[(\bar{x}) \pm KR]$ shall be calculated from the relevant test results (*see* **4.7.1.4**). If the values so obtained lie between the two specification limits, the lot shall be declared as conforming to the requirements of that characteristic.

4.7.1.4 The value of the factor K referred to in **4.7.1.1** to **4.7.1.3** shall be chosen in accordance with Table 2 given below, depending upon the acceptable quality level, namely, the percentage of non-conforming containers that could reasonably be tolerated.

Table 2 Values of K for Achieving Different Acceptable Quality

(*Clause* 4.7.1.4)

Acceptable Quality Level	Value of *K*
Not more than 3 percent defectives	0.4
Not more than 1.5 percent defectives	0.5
Not more than 0.5 percent defectives	0.6

4.7.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics determined on the composite sample, the test results for each of the characteristic shall satisfy the relevant requirements given in the product specification.

Examples:

In case of oil of peppermint, according to IS 528, total alcohols as menthol (percent by mass) is to be tested on individual samples and the value of *K* has been chosen as 0.4. In the case of oil of pine, according to IS 5757, distillation yield (percent by volume) and total alcohols as terpineol (percent by mass) are to be tested on individual samples and the value of *K* is 0.5. The conformity of the lot to the specification requirement in these two cases shall be judged as follows:

Product	*Indian Standard*	*Characteristic*	*Type*	*Specification Requirements*	*Criteria for Conformity*
Oil of peppermint	IS 528	Total alcohols as menthol, percent by mass	1	65, *Min*	$(\bar{x} - 0.4R) \geq 65$
		Total alcohols as menthol, percent by mass	2	45, *Min*	$(\bar{x} - 0.4R) \geq 45$
Oil of pine	IS 5757	Distillation yield, percent by volume	Below 185ºC	5.0, *Max*	$(\bar{x} + 0.5R) \geq 70$
			Below 200ºC	25.0, *Max*	$(\bar{x} + 0.5R) \geq 70$
			Below 230ºC	95.0, *Min*	$(\bar{x} - 0.5R) \geq 70$
		Total alcohols as terpineol, Percent by mass	–	70, *Min*	$(\bar{x} - 0.5R) \geq 70$

4.8 Packing and Labelling

Representative samples shall be packed in hermetically stoppered containers which shall be fastened and sealed with the seals of the owner and the sampler.

4.8.1 The precautions listed under **4.2.1** shall be observed.

4.8.2 All containers shall bear labels showing at least the following information to guarantee the authenticity and identity of the sample:

a) Sample number;
b) Nature and quantity of the product;
c) Name of the owner or his authorized representative;
d) Date of sampling;
e) Number, kind and marking of the containers;
f) Signatures and names and, if necessary addresses of the interested parties or their authorized representatives; and
g) Signature and name of the sampling supervisor.

4.9 Instructions regarding Costly Materials

Small containers are generally used for packing costly products.

4.9.1 The bulk sampling depends on the number of containers used; the combined partial samplings, however, shall not exceed the quantities necessary for a normal analysis. The interested parties shall agree in advance as to the size of the bulk sample and the manner in which it shall be done.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Fragrance and Flavour Sectional Committee, PCD 18

Organization	*Representative(s)*
CSIR-Central Institute of Medical and Aromatic Plants, Lucknow	Dr Prabodh K. Trivedi (***Chairman***)
All India Agarbathi Manufacturers Association, Bengaluru	Shri Sarath Babu P. S.
Aroma Sales Corporation, New Delhi	Shri Sunil Kumar Jain
CKC Fragrance and Flavours Private Limited, Kolkata	Shri Rishab Kothari Shri Chandrakant Kothari (*Alternate*)
CSIR-Central Food Technological Research Institute, Mysore	Shri Giridhar P. Shri Nagarajan S. (*Alternate*)
CSIR-Indian Institute of Integrative Medicine, Jammu	Shri Rajneesh Anand
CSIR-Indian Institute of Toxicology Research, Lucknow	Dr Alok Dhawan Shri Somendu Kumar Roy (*Alternate* I) Dr Sheelendra Pratap Singh (*Alternate* II)
CSIR-Institute of Himalayan Bio-Resource Technology, Palampur	Dr Vijai Kant Agnihotri
CSIR-North East Institute of Science and Technology, Jorhat	Dr Samit Chattopadhyay Dr Mohan Lal (*Alternate*)
CSIR-Central Institute of Medical and Aromatic Plants, Lucknow	Shri Sudeep Tandon Shri Chandan S. Chanotiya (*Alternate*)
Central Drugs Standard Control Organization, New Delhi	Dr. S. P. Shani
Central Drugs Testing Laboratory, Chennai	Shrimati C. Vijayalakshmi
Central Revenue Control Laboratory, New Delhi	Shri Suneel Mathur Shri Praful Dalal (*Alternate*)
Centre for Aromatic Plants, Dehradun	Shri Nirpendra K. Chauhan Ms Hema Lohani (*Alternate*)
Concert Trust, Consumers Association of India, Chennai	Representative
Consumer Voice, New Delhi	Shri B. K. Mukhopadhyay
D.V. Deo Industries, Cochin	Shri Aditya Deo
Directorate of Marketing and Inspection, Faridabad	Dy Agril Marketing Adviser
Drugs Control Organization, Jaipur, Rajasthan	Representative
Essential Oil Association of India, Delhi	Shri Ajay K. Jain Shri Pradeep Kumar Jain (*Alternate*)
Fab Flavours and Fragrances Private Limited, Delhi	Shri Gurnish Singh
Forest Research Institute (FRI), Dehradun	Shri V. K. Varshney
Fragrance and Flavour Development Centre, Kannauj	Shri S. V. Shukla Shri Nadeem Akbar (*Alternate*)

Organization	*Representative(s)*
Fragrances and Flavours Association of India, Mumbai	Shri Rishabh C. Kothari Shri Jaideep Gandhi (*Alternate*)
Givaudan India Private Limited, Mumbai	Shri Ajit Pal Shri Arshdeep K. Joshi (*Alternate*)
ITC Life Sciences and Technology Centre, Bengaluru	Dr James Bhaskar Shri Gurubasavaraja K. M. (*Alternate* I) Dr Vijayan Padmanabhan (*Alternate* II)
Indian Beauty and Hygiene Association, Mumbai	Ms Malathi Narayanan Ms Veena Balgi (*Alternate*)
Indian Pharmacopoeia Commission, Ghaziabad	Dr Jai Prakash Dr Manoj Kumar Pandey (*Alternate*)
Indian Society of Cosmetic Chemists, Mumbai	Dr Renuka Thergaonkar Ms Monisha Mullick (*Alternate*)
Jagat Aroma Oils Distillery, Kannauj	Shri Pradeep Kapoor Shri Vibhu Kapoor (*Alternate*)
Karnataka Soaps and Detergents Limited, Bengaluru	Shri A. E. Shankare Gowda Dr Mokashi (*Alternate*)
L Liladhar and Company, Mumbai	Shri Madhusoodan Mody
Lalji Aromatics Private Limited, Lucknow	Shri Rahul Mehrotra
Ministry of Micro, Small and Medium Enterprises, New Delhi	Shri L. B. S. Yadav
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Rakesh Sandal Industries, Kanpur	Shri Yogesh Dubey
S H Kelkar and Company Private Limited, Mumbai	Shri Amit Gulati Shri Avani Mainkar (*Alternate*)
SGS India Private Limited, Mumbai	Representative
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Ultra International Limited, Ghaziabad	Shri Sant Sanganeria Shrimati Bhuvana Nageshwaran (*Alternate*)
Voluntary Organisation in Interest of Consumer Education (VOICE), New Delhi	Shri M. A. U. Khan
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In Personal Capacity	Shri V. S. Venkatesha Gowda
BIS Director General	Shrimati Nagamani T., Scientist 'E' and Head (PCD) [Representing Director General (*Ex-officio*)]

Member Secretary

Shrimati D. Uma
Scientist 'D', (PCD), BIS

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BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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